PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2002-241813

(43)Date of publication of application : 28.08.2002

(51)Int.Cl. B22F 9/24
B01J 13/00

(21)Application number : 2001-044241

(22)Date of filing : 20.02.2001

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(54) METHOD FOR PRODUCING METALLIC COLLOIDAL LIQUID

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a method for producing a metallic colloidal liquid which exhibits stable dispersibility even in the change of pH, the presence of an electrolyte or the change of an atmospheric temperature, exhibits high dispersion stability even when the rate of change in temperature is high, or there is a temperature cycle, and in which the content of organic matters is also low as possible, and the characteristics of metallic fine particles such as electrical conductivity can be made the most of.

SOLUTION: A solution containing tannic acid and a solution containing the inorganic acid salt of a metal whose ionization series is nobler than that of hydrogen are mixed to produce a metallic colloidal liquid. On the mixing, both the viscosity of the solution containing tannic acid and that of the solution containing the inorganic acid salt of the metal whose ionization series is nobler than that of hydrogen measured by a B type viscosimeter are ≤300 mPa.s. Also, the volume ratio between the solution containing tannic acid and the solution containing the inorganic acid salt of the metal whose ionization series is nobler than that of hydrogen is 1/1 to 500/1 (the solution containing tannic acid/the solution containing the inorganic acid salt of the metal whose ionization series is nobler than that of hydrogen).

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CLAIMS

[Claim(s)]
[Claim 1]It is how to mix a solution characterized by comprising the following containing tannic acid, and a solution in which an ionization series contains an inorganic acid salt of **** metal from hydrogen, and to manufacture metallic colloid liquid, and is at the mixed time.
A solution in which viscosity which a solution containing said tannic acid and said ionization series measured by a Brookfield viscometer of a solution which contains an inorganic acid salt of **** metal from hydrogen is both 300 or less mPa-s, and contains said tannic acid.
An inorganic acid salt of metal more **** than hydrogen in said ionization series.

[Claim 2]A manufacturing method of the metallic colloid liquid according to claim 1, wherein initial temperature of a solution in which a solution and an ionization series containing tannic acid contain an inorganic acid salt of **** metal from hydrogen at the time of mixing is both 5-75 ** and agitating speed under mixing is 30-2000 rpm.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the manufacturing method of metallic colloid liquid.
[0002]
[Description of the Prior Art]Although metal microscopic particles are distributing to water, an organic solvent, etc., metallic colloid liquid is very unstable thermodynamically, and with time, metal microscopic particles condense it and are in the sedimenting tendency. When the case where an electrolyte exists, and pH change, it is remarkable, and it is remarkable also when ambient temperature changes, and it has a case where especially the rate of change in temperature is large, and a temperature cycle. Such a tendency becomes remarkable also when metallic colloid liquid is high concentration.
[0003]Although such metallic colloid liquid is known for many years, For example, to JP,10-66861,A. After managing temperature, make a silver nitrate aqueous solution and ferrous-citrate solution react under 2000-6000-rpm stirring, and the colloid liquid of a silver particulate is obtained, Obtaining the silver colloid liquid used for the coating material for transparent conducting film formation is indicated by by adding sodium nitrate solution to the solid part obtained by sedimenting, removing iron, and also centrifuging by the gravity of 3000G, obtaining silver solid content, and carrying out re dispersion of it to water. To JP,2000-87122,A. In order to compensate the fault of the art indicated in the above-mentioned gazette, it prepares in the atmosphere which does not contain oxygen substantially, and obtaining the mixed colloid liquid of the silver colloid liquid or silver similarly used for the coating material for transparent conducting film formation, and palladium is indicated. To JP,11-80647,A, obtaining the precious metals or the copper colloid liquid which uses for a color material with high chroma saturation the amount pigment agent of polymers in which a number average molecular weight has a specific structure of 1000-1 million by using it as a polymers system dispersing agent is indicated.
[0004]Although the above-mentioned various methods were excellent as metallic colloid liquid used for a specific use, when high dispersion stability is required, or when making the

characteristic of metal microscopic particles notably conspicuous and using for a catalyst, a conductive material, etc., it became clear that there was a big problem.

[0005]The metal microscopic particles in the metallic colloid liquid obtained by the method indicated to JP,10-66861,A or JP,2000-87122,A have the carboxylate ion originating in citrate on the surface, and are distributing it by the electric rebounding. Therefore, JP,10-66861,A has the necessity of flushing iron from the metallic colloid liquid generated with the art of the statement, and if an electrolyte is added or pH is changed so that this may also show, metal microscopic particles will carry out coagulation. Therefore, when using it as a catalyst in the system containing an electrolyte, and concentration of the system of reaction was made high, it became clear that the metal microscopic particles in metallic colloid liquid carried out coagulation, and the desired characteristic was not obtained. Since the surface activity of metal microscopic particles could not use enough but contact between particles was barred when using it as a catalyst, since it had such ion on the surface, it also became clear that it could not use as a high electrical conducting material.

[0006]In the colloidal solution of the precious metals or copper obtained by the method indicated to JP,11-80647,A. Since it is made to distribute by making it get twisted around the surface of metal microscopic particles by using the amount pigment agent of polymers as protective colloid, By a temperature change, the interaction of polymers and metal microscopic particles arises and it precipitates, It became clear from it being necessary to make a lot of polymers stick to a surface of metal, in order to make it distribute effectively that there was a problem in using for the catalyst using the surface activity of metal microscopic particles and the application which contacts metal microscopic particles and has about the same volume resistance value as metal.

[0007]

[Problem(s) to be Solved by the Invention]This invention shows the dispersibility stable also by change of pH, existence of an electrolyte, and change of ambient temperature in view of the above, Even when it has a case where especially the rate of change in temperature is large, and a temperature cycle, high dispersion stability is shown, and there are as much as possible few organic matters, and they aim at providing the manufacturing method of the metallic colloid liquid which can employ the characteristic of metal microscopic particles, such as conductivity, efficiently.

[0008]

[Means for Solving the Problem]In [this invention is the method of mixing a solution containing tannic acid and a solution containing an inorganic acid salt of metal more **** than hydrogen in an ionization series, and manufacturing metallic colloid liquid, and] the time of mixing, A solution in which viscosity measured by a Brookfield viscometer of a solution in which a solution and the above-mentioned ionization series containing the above-mentioned tannic acid contain an inorganic acid salt of **** metal from hydrogen is both 300 or less mPa-s, and contains the above-mentioned tannic acid, A volume ratio with a solution in which the above-mentioned ionization series contains an inorganic acid salt of **** metal from hydrogen is a manufacturing method of metallic colloid liquid which is 1 / 1 - 500/1 (solution in which the

solution/ionization series containing tannic acid contain an inorganic acid salt of **** metal from hydrogen). This invention is explained in full detail below.

[0009]A result to which this invention person carried out various examination about manufacture of metallic colloid liquid, By using what is generally called tannic acid (otherwise, expressed as gallotannic acid, nutgall tannin, etc.), and manufacturing metallic colloid liquid under specific conditions, It found out that metallic colloid liquid in which dispersion stability which was excellent even if it did not use a dispersing agent is shown was producible, and resulted in this invention.

[0010]Production of metallic colloid liquid by chemicals reduction known until now had indispensable use of protective colloid which consists of a dispersing agent and various high molecular compounds like sodium acid citrate. However, according to this invention, even if it uses neither a dispersing agent nor protective colloid, metallic colloid liquid with high dispersion stability is producible, And even when dispersibility stable also by change of pH, and existence of an electrolyte and change of ambient temperature was shown and it had a case where especially a rate of change in temperature is large, and a temperature cycle, it turned out that metallic colloid liquid with few organic matters in which high dispersion stability is shown is producible.

[0011]J.W. To Slot, H.J.Geuze, European Journalof Cell Biology, 38, and 87-93 (1985). Although a chlorauric acid solution and a solution which dissolved tannic acid, trisodium citrate, and potassium carbonate were mixed and producing gold colloid liquid was indicated, obtained gold colloid liquid had golden concentration very as thin as 0.58 g/L. Tannic acid of per [golden ion univalent / g] at this time was 0.00057-0.0099g, and tannic acid of per trisodium citrate 1g was 0.049-0.85g. As a result of this invention person's examining this method, even if it made golden concentration high, using this method as it is, it became clear that colloid liquid with high dispersion stability could not be obtained.

[0012]H. To Muhlpford, Experientia, and 38,1127-1128 (1982), if only a tannic acid solution or a citric acid solution is used, it is indicated that gold colloid liquid is unproducible.

[0013]When this invention person examined combination and manufacturing conditions to these, even if it was tannic acid independent, it found out that an outstanding metallic colloidal solution could be produced. This invention is the method of mixing a solution containing tannic acid and a solution containing an inorganic acid salt of metal more **** than hydrogen in an ionization series, and manufacturing metallic colloid liquid.

[0014]As for an addition of the above-mentioned tannic acid, it is preferred that they are 0.01-6g to metal ion univalent / g. That is, for example, in the case of a univalent silver ion, additions of tannic acid of per silver ion 1g are 0.01-6g, and, in the case of trivalent golden ion, additions of tannic acid of per golden ion 1g are 0.03-18g.

[0015]Since it is it hard to follow a reduction reaction of a metal ion that an addition of tannic acid is less than 0.01g to metal ion univalent / g, Since a conversion thing of superfluous tannic acid or tannic acid will stick to a surface of metal if it becomes difficult to obtain desired metallic colloid liquid and exceeds 6 g, dispersion stability is not influenced, but organic substance quantity increases and it is not desirable.

[0016]As **** metal, the above-mentioned ionization series can mention gold, silver, copper, platina ** palladium, rhodium, iridium, osmium, a ruthenium, a rhenium, etc. from hydrogen, for example. The above-mentioned ionization series is not limited especially as an inorganic acid salt of **** metal from hydrogen, for example, a nitrate of the various above-mentioned metal, nitrite salt, a chloride, an oxide, a perchlorate, etc. can be mentioned. These inorganic acid salts may be used independently and two or more sorts may be used together. In this invention, the above-mentioned metal must be used as not a form but mineral salt of organic salt. Since the organic matter will stick to a surface of metal if it uses in a form of organic salt, a content of an organic matter becomes large and the characteristic of metal microscopic particles, such as conductivity, is checked.

[0017]this invention person found out that a solution containing tannic acid and an ionization series influenced greatly viscosity in a case of mixing a solution containing an inorganic acid salt of metal more **** than hydrogen, and character of metallic colloid liquid in which a capacity factor is obtained.

[0018]Viscosity which a solution and an ionization series containing tannic acid measured from hydrogen by a Brookfield viscometer of a solution containing an inorganic acid salt of **** metal at the time of mixing a manufacturing method of metallic colloid liquid of this invention, It is characterized by being both 300 or less mPa-s, and volume ratios of a solution containing tannic acid and a solution in which an ionization series contains an inorganic acid salt of **** metal from hydrogen being 1 / 1 - 500/1 (solution in which the solution/ionization series containing tannic acid contain an inorganic acid salt of **** metal from hydrogen).

[0019]When not fulfilling this condition, a contact opportunity of tannic acid in inside of mixed liquor and a metal ion may decrease, and desired metallic colloid liquid may not be obtained, but very insufficient metallic colloid liquid of dispersion stability may be obtained to a pH change, a temperature change, etc.

[0020]this invention person also found out that mixing requirements in a case of mixing a solution containing an inorganic acid salt of metal more **** than hydrogen were also large in character of metallic colloid liquid obtained, and a solution and an ionization series containing tannic acid influenced.

[0021]As for a manufacturing method of metallic colloid liquid of this invention, at the time of mixing, it is preferred that initial temperature of a solution in which a solution and an ionization series containing tannic acid contain an inorganic acid salt of **** metal from hydrogen is both 5-75 **, and agitating speed under mixing is 30-2000 rpm.

[0022]Since tannic acid and a metal ion will not react uniformly if temperature is less than 5 ** and agitating speed is less than 30 rpm, character of metallic colloid particles changes and metallic colloid liquid with very bad dispersion stability may be obtained to a pH change, a temperature change, etc. On the other hand, when agitating speed exceeds 2000 rpm exceeding 75 ** in temperature, reaction velocity becomes quick too much and metallic colloid liquid in which metallic colloid particles sedimented may be obtained from the beginning. If temperature exceeds 75 **, degradation of a solution containing tannic acid will become quick.

[0023]Metallic colloid liquid obtained by this invention may reveal sufficient dispersion stability

and the characteristics, such as good conductivity, even if a reducing agent or a dispersing agent of further others are not added, but. In a manufacturing method of metallic colloid liquid of this invention instead of what eliminates use of other reducing agents or a dispersing agent, a process of adding a reducing agent and a dispersing agent of further others may be established.

[0024]It is not limited especially as the above-mentioned reducing agent, for example, ferrous sulfate, formalin, alcohol, hydrogen gas, phenidone, Metol, tartaric acid, amines, etc. can be mentioned. It is not limited especially as the above-mentioned dispersing agent, for example, polymer, such as citrate; malic acid, such as citrate and sodium acid citrate, salt; polyvinyl alcohol of those, polyethylene-glycol polyacrylamide, polyethyleneimine, gum arabic, and gelatin, etc. can be mentioned.

[0025]A catalyst [in / in metallic colloid liquid obtained by this invention / an oxidation-reduction reaction or a photocatalysis], Conductive ink besides [which has the high conductivity of a minute globular form base material and the metal average] a conductive material, It can use for various uses which can employ the feature of metal microscopic particles with few organic matter contents efficiently, such as a color material using coloring of an optical material, a transparent conductive material, antistatic materials, a charge of an electromagnetic wave shielding material, and metallic colloid liquid.

[0026]By controlling a ratio with a charge, concentration, temperature, agitating speed, and other additive agents, and storing a reaction condition in a specific range further, using tannic acid, so that more clearly than the above. Dispersibility stable also by change of pH, and existence of an electrolyte and change of ambient temperature was shown, and even when it had a case where especially a rate of change in temperature is large, and a temperature cycle, it became possible to provide metallic colloid liquid in which high dispersion stability is shown. Since an addition of tannic acid to a metal ion can be extremely managed with a small quantity, it becomes possible to provide metallic colloid liquid which can employ the characteristic of metal microscopic particles efficiently.

[0027]

[Example]Although an example is hung up over below and this invention is explained to it in more detail, this invention is not limited only to these examples.

[0028](Example 1) 0.5 g of tannic acid (the Wako Pure Chemical Industries, Ltd. make, for chemicals) was dissolved in the ion exchange water of 100mL (2 or less mPa-s of viscosity, A liquid). Next, having used the magnetic stirrer under the room temperature and stirring at 500 rpm, solution (2 or less mPa-s of viscosity, B liquid) 2mL containing 1 g of silver nitrate (the Wako Pure Chemical Industries, Ltd. make, special grade chemical) was made dropped, and silver colloid liquid was produced. Production of silver colloid liquid was performed at the room temperature. At this time, tannic acid to the silver ion 1g will be set to 0.79 g, and a stoichiometrical metal generated amount will be 6.2 g/L. The mixture ratio of A liquid and B liquid will be 50/1. Measurement of the viscosity of A liquid and B liquid was performed using BM type viscosity meter by TOKIMEC, INC. The measurement minimums which can trust the opportunity are 10 mPa-s.

[0029]The following evaluations were performed about the obtained metallic colloid liquid. (Dispersibility evaluation) After stirring metallic colloid liquid well, optimum dose was moved to the test tube of capacity 30mL, and it was neglected under the following condition. and -- if solid content sediments -- x -- if it did not sediment, it was estimated as O. The experiment was conducted on the following conditions.

[0030]pH: 2 (chloride adjusts) and 12 (sodium hydroxide solution adjusts)
Electric conductivity: 1 mS/cm and 10 mS/cm (sodium hydroxide solution adjusts)
Temperature: A room temperature (about 20 **) and 80 ** (the ion exchange water warmed when a heater adjusted and volume became less was added slowly)
Heat cycle test: Carry out sudden heating at 80 **, quench at 10 ** in a 1-hour neglect -> refrigerator, and it is two-cycle ****** about 1-hour neglect. The ion exchange water warmed when volume became less also in this case was added slowly.

[0031](Organic-substance-quantity measurement) Metallic colloid liquid is put into a suitable container, and natural seasoning was carried out until it became a constant mass in a desiccator. TG/DTA300 by the SEIKO electronic industry company was used for the natural seasoning thing, and it asked for the thermo gravity change in the atmosphere from a room temperature to 500 ** by a part for heating-rate/of 10 **. And the weight loss from 100 ** to 500 ** was calculated.

[0032](Example 2) It carried out like Example 1 except the quantity of tannic acid having been 0.007 g. Tannic acid to the silver ion 1g is set to 0.01 g at this time.

[0033](Example 3) It carried out like Example 1 except having dissolved the tannic acid 3.8g in the ion exchange water of 500mL. Tannic acid to the silver ion 1g will be set to 6 g at this time, and the mixture ratio of A liquid and B liquid will be 250/1.

[0034](Example 4) It carried out like Example 1 except solution (2 or less mPa-s of viscosity) 100mL containing 1 g of silver nitrate having been dropped. At this time, a stoichiometrical metal generated amount will be 3.2 g/L. The mixture ratio of A liquid and B liquid will be 1/1.

[0035](Example 5) It carried out like Example 1 except having dissolved the tannic acid 0.5g in the ion exchange water of 1000mL (2 or less mPa-s of viscosity). At this time, a stoichiometrical metal generated amount will be 0.63 g/L. The mixture ratio of A liquid and B liquid will be 500/1.

[0036](Example 6) It carried out like Example 1 except having dissolved the tannic acid 0.5g in thing 100mL which mixed ion exchange water and glycerin (the Wako Pure Chemical Industries, Ltd. make, special grade chemical), and made Brookfield viscometer viscosity 300 mPa-s beforehand.

[0037](Example 7) After adjusting both A liquid and B liquid to 5 **, it carried out like Example 1 except having mixed agitating speed as 30 rpm.

[0038](Example 8) After adjusting both A liquid and B liquid to 75 **, it carried out like Example 1 except having mixed agitating speed as 2000 rpm.

[0039](Example 9) It carried out like Example 1 except having dissolved the tannic acid 0.5g in thing 100mL (2 or less mPa-s of viscosity) which mixed ion exchange water and methyl alcohol (the Wako Pure Chemical Industries, Ltd. make, special grade chemical) to 1 to 1 by the

capacity factor beforehand.

[0040](Example 10) In addition to the tannic acid 0.5g, 46 g of ferrous sulfate 7 hydrates (the Wako Pure Chemical Industries, Ltd. make, special grade chemical) were performed like Example 1 except having dissolved in the ion exchange water of 100mL (2 or less mPa-s of viscosity). At this time, the weight ratio of tannic acid to ferrous sulfate is set to 0.02.

[0041](Example 11) The silver colloid liquid produced in Example 1 was put into the Kurabo Industries, Ltd. make and Seng Tori Katt U-10, the centrifuge performed the ultrafiltration for [3000 rpmx] 30 minutes, and capacity was eventually set to 5mL. At this time, a stoichiometrical metal generated amount will be 127 g/L. The obtained concentration silver colloid liquid was similarly estimated as Example 1.

[0042](Example 12) In addition to the tannic acid 0.5g, 1.1 g of sodium-acid-citrate dihydrate (the Wako Pure Chemical Industries, Ltd. make, special grade chemical) was performed like Example 1 except having dissolved in the ion exchange water of 100mL. At this time, the weight ratio of tannic acid to sodium acid citrate is set to 0.5.

[0043](Example 13) It carried out like Example 1 except having used solution 2mL which contains 1 g of chloroauric acid 4 hydrates (the Wako Pure Chemical Industries, Ltd. make, special grade chemical) instead of a silver nitrate aqueous solution in Example 1. At this time, tannic acid to the golden ion 1g will be set to 0.1 g (per [g / univalent// 0.33g]), and a stoichiometrical metal generated amount will be 4.6 g/L.

[0044](Example 14) It carried out like Example 1 except having used solution 2mL which contains 1 g of cupric nitrate acid hydrates (the Wako Pure Chemical Industries, Ltd. make, best) instead of a silver nitrate aqueous solution in Example 1. At this time, tannic acid to the copper ion 1g will be set to 1.9 g (per [g / univalent// 0.95g]), and a stoichiometrical metal generated amount will be 4.6 g/L.

[0045](Comparative example 1) It carried out like Example 1 except having dissolved the tannic acid 6.3g in the ion exchange water of 1000mL. At this time, tannic acid to the silver ion 1g will be set to 10 g, and the mixture ratio of A liquid and B liquid will be 500/1.

[0046](Comparative example 2) It carried out like Example 1 except solution (2 or less mPa-s of viscosity, B liquid) 200mL containing 1 g of silver nitrate having been dropped, and having produced silver colloid liquid. At this time, the mixture ratio of A liquid and B liquid will be 0.5/1.

[0047](Comparative example 3) It carried out like Example 1 except having dissolved the tannic acid 0.5g in the ion exchange water of 2000mL. At this time, the mixture ratio of A liquid and B liquid will be 1000/1.

[0048](Comparative example 4) It carried out like Example 1 except having dissolved the tannic acid 0.5g in solution 100mL which mixed ion exchange water and glycerin and made Brookfield viscometer viscosity 400 mPa-s beforehand.

[0049](Comparative example 5) After adjusting both A liquid and B liquid to 5 **, it carried out like Example 1 except having mixed agitating speed as 5 rpm.

[0050](Comparative example 6) After adjusting both A liquid and B liquid to 75 **, it carried out like Example 1 except having mixed agitating speed as 3000 rpm.

[0051](Comparative example 7) It carried out like Example 1 except having dissolved the ferrous sulfate 7 hydrate 0.5g in the ion exchange water of 100mL instead of tannic acid.
[0052](Comparative example 8) Instead of tannic acid, it carried out like Example 1 except having dissolved 6.4 g of sodium-acid-citrate dihydrate (the Wako Pure Chemical Industries, Ltd. make, special grade chemical), and the ferrous sulfate 7 hydrate 5.5g in the ion exchange water of 100mL.
[0053](Comparative example 9) The silver colloid liquid produced by the comparative example 8 is put into the Kurabo Industries, Ltd. make and Seng Tori Katt U-10, The centrifuge performed the ultrafiltration for [3000 rpmx] 30 minutes, when amount of water became less, it carried out by adding ion exchange water and repeating an ultrafiltration, and eventually, electric conductivity (it measures by Toa Electronics, Ltd. make and CM-20S) was made into 100microS/cm, and capacity was set to 100mL.
[0054](Comparative example 10) It carried out like Example 1 except having dissolved SORUSU pass 27000 (made by ABISHIA) 4.0g and the ferrous sulfate 7 hydrate 5.5g which are polymers system pigment agents instead of in the ion exchange water of 100mL. [tannic acid] At this time, the SORUSU pass 27000 to the silver ion 1g is set to 6.3 g. The above result was shown in Table 1.
[0055]
[Table 1]

	分散安定性							有機物
	pH		電導度 (mS／cm)		温度 (℃)		温度サイクル試験	重量損失 (重量%)
	2	12	1	10	室温	80		
実施例1	○	○	○	○	○	○	○	15
実施例2	○	○	○	○	○	○	○	11
実施例3	○	○	○	○	○	○	○	20
実施例4	○	○	○	○	○	○	○	18
実施例5	○	○	○	○	○	○	○	11
実施例6	○	○	○	○	○	○	○	12
実施例7	○	○	○	○	○	○	○	17
実施例8	○	○	○	○	○	○	○	20
実施例9	○	○	○	○	○	○	○	14
実施例10	○	○	○	○	○	○	○	14
実施例11	○	○	○	○	○	○	○	13
実施例12	○	○	○	○	○	○	○	20
実施例13	○	○	○	○	○	○	○	13
実施例14	○	○	○	○	○	○	○	10
比較例1	○	○	○	○	○	×	×	35(多い)
比較例2	×	×	○	×	○	×	×	20
比較例3	×	×	○	×	○	×	×	23
比較例4	○	○	○	○	○	×	×	30(多い)
比較例5	×	×	×	×	×	×	×	18
比較例6	×	×	○	×	○	×	×	30(多い)
比較例7	×	×	×	×	×	×	×	18
比較例8	×	×	×	×	×	×	×	35(多い)
比較例9	×	×	×	×	○	×	×	23
比較例10	○	×	○	×	○	×	×	78(多い)

[0056]

[Effect of the Invention]It consists of above-mentioned composition in this invention. Therefore, change of pH, and existence of an electrolyte and change of ambient temperature also show high dispersibility, The metallic colloid liquid which shows high dispersion stability also under conditions with the case where especially the rate of change in temperature is large, or a temperature cycle, and there are few organic matters to which it is sticking, and can employ the characteristic of metal microscopic particles efficiently can be provided.

[Translation done.]